

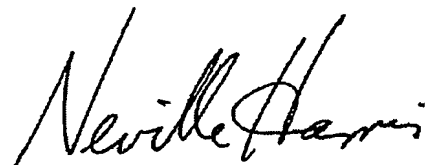
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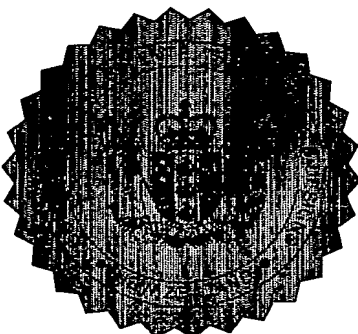
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Dated 31 July 2003.

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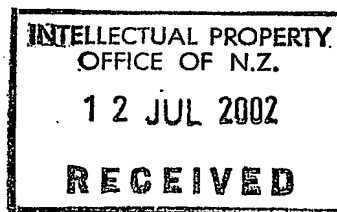


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**PATENTS FORM NO. 4**

Appln Fee: \$50.00

James &amp; Wells ref: 27039/16 CL

**PATENTS ACT 1953**  
**PROVISIONAL SPECIFICATION****COMPOUND PREPARATION METHOD**

We, Celentis Limited, a New Zealand company of East Street, Ruakura Campus, Hamilton, New Zealand, do hereby declare this invention to be described in the following statement:

## COMPOUND PREPARATION METHOD

### TECHNICAL FIELD

This invention relates to a method of preparing a compound. Preferably the compound involved may be prepared for subsequent analysis of its components. However, in  
5 other embodiments, such compounds may be prepared with a view to providing a reactant for other processes. The present invention may preferably allow a compound to be rendered into a plurality of substantially homogenous sized particles.

### BACKGROUND ART

Some types of chemical analysis equipment require a sample for analysis to be  
10 supplied as a number of substantially homogenous particles. Furthermore, some types of chemical reactions can also require one of the reactant compounds involved again to be supplied as a plurality of particles of substantially the same size.

An existing technique used to prepare such samples or compounds employs a grinding procedure. If the compound involved is wet or composed of organic tissue, it will  
15 need to be thoroughly dried prior to grinding.

The need to grind thoroughly and also dry such compounds makes the preparation method employed relatively slow. Having to both dry and also grind a compound is both slow and relatively laborious work.

Furthermore, as the compound preparation time increases, so do the chances of the  
20 compound being exposed to some form of contaminant, or alternatively degrading with age.

This type of preparation work normally must also be completed within a laboratory environment. This again puts some limitations on the utility of such preparation

methods, which cannot be employed out in the field where (for example) a sample has been freshly collected.

One application where an improved method of compound preparation would provide advantages is in the use of near infra-red (NIR) spectrophotometers. These devices  
5 can detect the presence and also the concentration of a wide range of analytes compounds within a properly prepared sample. In addition, the analytes, which can be targeted by NIR spectrophotometers, encompass a wide range of compounds present in organic tissues, and as such, a preparation method which could quickly prepare an organic sample for analysis would be of advantage.

10 An improved method of preparing a compound which addressed any or all of the above issues would be of advantage. A method which could render a compound into a plurality of substantially homogenous size particles quickly without the need for expensive or complicated equipment, or a laboratory environment, would be of advantage.

15 All references, including any patents or patent applications cited in this specification are hereby incorporated by reference. No admission is made that any reference constitutes prior art. The discussion of the references states what their authors assert, and the applicants reserve the right to challenge the accuracy and pertinency of the cited documents. It will be clearly understood that, although a number of prior art  
20 publications are referred to herein, this reference does not constitute an admission that any of these documents form part of the common general knowledge in the art, in New Zealand or in any other country.

It is acknowledged that the term 'comprise' may, under varying jurisdictions, be attributed with either an exclusive or an inclusive meaning. For the purpose of this  
25 specification, and unless otherwise noted, the term 'comprise' shall have an inclusive meaning - i.e. that it will be taken to mean an inclusion of not only the listed

components it directly references, but also other non-specified components or elements. This rationale will also be used when the term 'comprised' or 'comprising' is used in relation to one or more steps in a method or process.

5 It is an object of the present invention to address the foregoing problems or at least to provide the public with a useful choice.

Further aspects and advantages of the present invention will become apparent from the ensuing description which is given by way of example only.

#### **DISCLOSURE OF INVENTION**

10 According to one aspect of the present invention there is provided a compound preparation method characterised by the steps of;

- (i) cooling the compound to increase its rigidity, and
- (ii) mechanically processing the compound to render same into a plurality of particles or components of substantially the same size, and
- (iii) subjecting the rendered compound to an analysis and/or reaction process.

15 According to a further aspect of the present invention there is provided a method of preparing a compound substantially as described above, wherein a compound is prepared to provide a sample for an analysis procedure.

20 According to yet another aspect of the present invention there is provided a method of preparing a compound substantially as described above, wherein the compound is cooled with liquid carbon dioxide.

According to a further aspect of the present invention there is provided a method of preparing a compound substantially as described above, wherein the compound is mechanically processed by at least one rotating blade.

According to yet another aspect of the present invention there is provided a compound preparation method substantially as described above, wherein the rendered compound is analysed using a near infra-red spectrophotometer.

5 According to yet another aspect of the present invention there is provided a compound preparation method substantially as described above wherein the compound is composed of or formed from plant tissue.

The present invention relates to an improved method of preparing a compound. Any number and range of different types of compounds may be prepared using the present invention depending on the particular application which it is employed within.  
10 However, it is envisioned that the preparation method discussed below could primarily be used to prepare a small volume or weight of compounds or samples.

Those skilled in the art should appreciate that after preparation the compound involved may be subjected to various further processing, analysis or reactions depending on the application which the present invention is used within.

15 Reference throughout this specification will also be made to the present invention being used to provide a sample preparation method where the sample involved is to be analysed to investigate its constituent components. The present invention may provide a preparation method which can allow a sample compound to be rendered into a plurality of distinct particles that are substantially the same size. However, those  
20 skilled in the art should appreciate that other applications are also envisioned for the present invention and reference to the above only throughout this specification should in no way be seen as limiting. For example, in one alternative embodiment, the present invention may be used to prepare a compound to be reacted with other materials.

25 In a further preferred embodiment the compound to be prepared may be organic in

nature, such as plant or animal tissue. Organic materials normally contain a high moisture content, and as such sample preparation time is relatively long using prior art preparation methods. However, through use of the present invention the time required to prepare such samples can be substantially reduced.

5 Reference throughout this specification will also be made to a sample prepared in accordance with the present invention being plant tissue. However, those skilled in the art should appreciate that other types of compounds or organic materials may also be prepared using the present invention, and reference to the above only throughout this specification should in no way be seen as limiting.

10 In a preferred embodiment the present invention may be used to prepare a sample for analysis by a near infra-red spectrophotometer. Near infra-red (NIR) spectrophotometers can detect the presence and also concentration of a wide variety of analytes, including those commonly found and of interest within organic materials such as plant tissue.

15 Preferably the first step employed in the method of the present invention is to cool the sample or compound to be prepared, thereby increasing its physical rigidity. The temperature of the compound may be lowered significantly, which in some instances will freeze the compound solid.

In a further preferred embodiment a sample may be exposed to a cooling agent to  
20 achieve the cooling effect required. Such a cooling agent may be a further compound which can be intimately exposed to a sample to cool same.

For example, in a preferred embodiment a cooling agent or material may be provided through the use of liquefied carbon dioxide. A sample may be dipped or immersed in liquefied carbon dioxide to rapidly reduce its temperature and therefore substantially  
25 increase its physical rigidity. The time required for the cooling to be completed is

relatively short with liquefied carbon dioxide, therefore providing a relatively short sample preparation time.

However, in alternative embodiments other means for cooling a sample may be employed. For example, in one alternative embodiment a sample may be immersed or  
5 dipped into liquid nitrogen, again to provide the cooling effect required. In yet another alternative embodiment a sample may be cooled using freeze drying equipment to again quickly reduce the temperature and increase the rigidity of the compound or sample.

Reference throughout this specification will however be made to a sample initially  
10 being cooled through immersion in liquefied carbon dioxide. However, those skilled in the art should appreciate that other types of cooling agents or equipment may also be employed and reference to the above only throughout this specification should in no way be seen as limiting.

Preferably after a sample has been cooled and its physical rigidity increased, it may  
15 then be subjected to a mechanical processing step. This mechanical processing can be used to render the sample into a plurality of particles or components which have substantially the same size. This in effect will homogenise the sample rendering it into a collection of particles or component pieces with a substantially uniform nature. The actual end product or final form of the rendered sample will be determined by the  
20 degree of mechanical processing employed in addition to the moisture content of the sample.

In a further preferred embodiment the mechanical processing step employed may be completed through use of a rotating blade. The cooled sample or compound may be placed within a container which also houses a blade adapted to be driven in a circular  
25 motion. When activated, the blade will make a large number of cuts through the material of the sample which has been temporarily stiffened through the cooling step



discussed above. The rotating blade employed can then shatter the relatively rigid sample to render same into a plurality of particles or portions of substantially the same size. This type of rotating blade apparatus can easily be provided through a common blender or food processor. Varying sizes of samples or compounds may also be  
5 processed depending on the capacity of the equipment used.

Reference throughout this specification will also be made to a sample or compound being mechanically processed through use of a rotating blade substantially as described above. However, those skilled in the art should appreciate that other types of mechanical processing systems may also be employed and reference to the above  
10 only throughout this specification should in no way be seen as limiting.

The present invention may provide many potential advantages over the prior art.

The combination of a cooling phase and mechanical processing phase allows a sample to be rendered into a plurality of substantially homogenous small particles which contain essentially the same moisture content as the sample at the start of the  
15 processing method. The moisture content already present within the sample allows analytes of interest to in effect remain "in solution", ready for presentation to the NIR spectrophotometer or other similar instrument.

Eliminating the need for drying further simplifies and speeds up the sample or compound preparation method provided. After mechanical rendering has been  
20 completed, a sample may then be directly presented to a spectrophotometer.

The present invention may also be used to quickly, easily and inexpensively prepare numerous different types of compounds for subsequent analysis or further reactions with other compounds. The present invention can be used to break up a compound into a large number of small, even or homogenous particles relatively quickly for use  
25 in a large number of applications.

Furthermore, the equipment or apparatus employed in conjunction with the present invention is readily portable and can be used in the field at sample or compound collection sites, to immediately prepare a sample for further analysis or reaction. This reduces the chances of the sample becoming contaminated through long periods of storage or handling and also reduces the chances of the sample degrading over time.

The use of liquefied carbon dioxide in preferred embodiments also allows a sample or compound to be cooled rapidly and effectively. Furthermore, liquefied carbon dioxide is a relatively inexpensive cooling material and is safer for operators to handle than liquid nitrogen at lower temperatures. Furthermore, the use of carbon dioxide is preferable where a sample to be prepared is to be analysed for its nitrogen content. Furthermore, carbon dioxide being higher temperature than liquid nitrogen will extend the lifespan of the equipment used in conjunction with the present invention. As the carbon dioxide employed is of a higher temperature, it degrades the equipment used slower than liquid nitrogen.

## 15 **BRIEF DESCRIPTION OF DRAWINGS**

Further aspects of the present invention will become apparent from the following description which is given by way of example only and with reference to the accompanying drawing in which:

20 **Figure 1** illustrates a block schematic diagram of the processes executed by a method of preparation in accordance with a preferred embodiment of the present invention;

## **BEST MODES FOR CARRYING OUT THE INVENTION**

Figure 1 illustrates a block schematic diagram of the steps executed in a method of preparation provided in accordance with a preferred embodiment.

In the instance discussed, the present invention is adapted to prepare a plant tissue sample for subsequent analysis by a near infra-red spectrophotometer.

The sample to be prepared is initially drawn in Step 0. For example, in a preferred instance a grass sample is cut from a paddock to provide the sample required.

5 In the schematic diagram shown, Step 1 is implemented to cool the sample provided and therefore increase its rigidity. Preferably this step is executed through immersing the sample in a container of liquefied carbon dioxide. This will snap-freeze the vegetative sample, substantially increasing the rigidity of the vegetative material, while also preserving the moisture content present within the vegetation.

10 After the sample has been retrieved from the carbon dioxide it then may be presented to a kitchen or household blender at Step 2 of the methodology executed. A blender with an associated rotating blade can be used to cut up and mechanically render the snap frozen vegetative sample into a plurality of distinct and substantially homogenised particles. A large number of relatively small particles of substantially  
15 the same size may be provided through this action. The increased rigidity of the frozen vegetation allows the rotating blade of the blender to shatter the vegetation into a collection of small particles.

Finally, once the cooling and subsequent mechanical processing Steps 1 and 2 are completed, the processed sample may be presented to a NIR spectrophotometer at Step

20 3. The sample can be directly presented to the spectrophotometer after mechanical processing Step 2. Through preserving the moisture content of the sample during the processing method executed, analytes of interest remain free for a spectrophotometer to detect same without a solvent being applied or used.

Aspects of the present invention have been described by way of example only and it should be appreciated that modifications and additions may be made thereto without departing from the scope thereof.

CELENTIS LIMITED

by its Attorneys



JAMES & WELLS

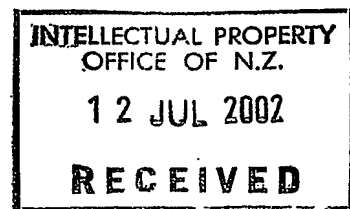
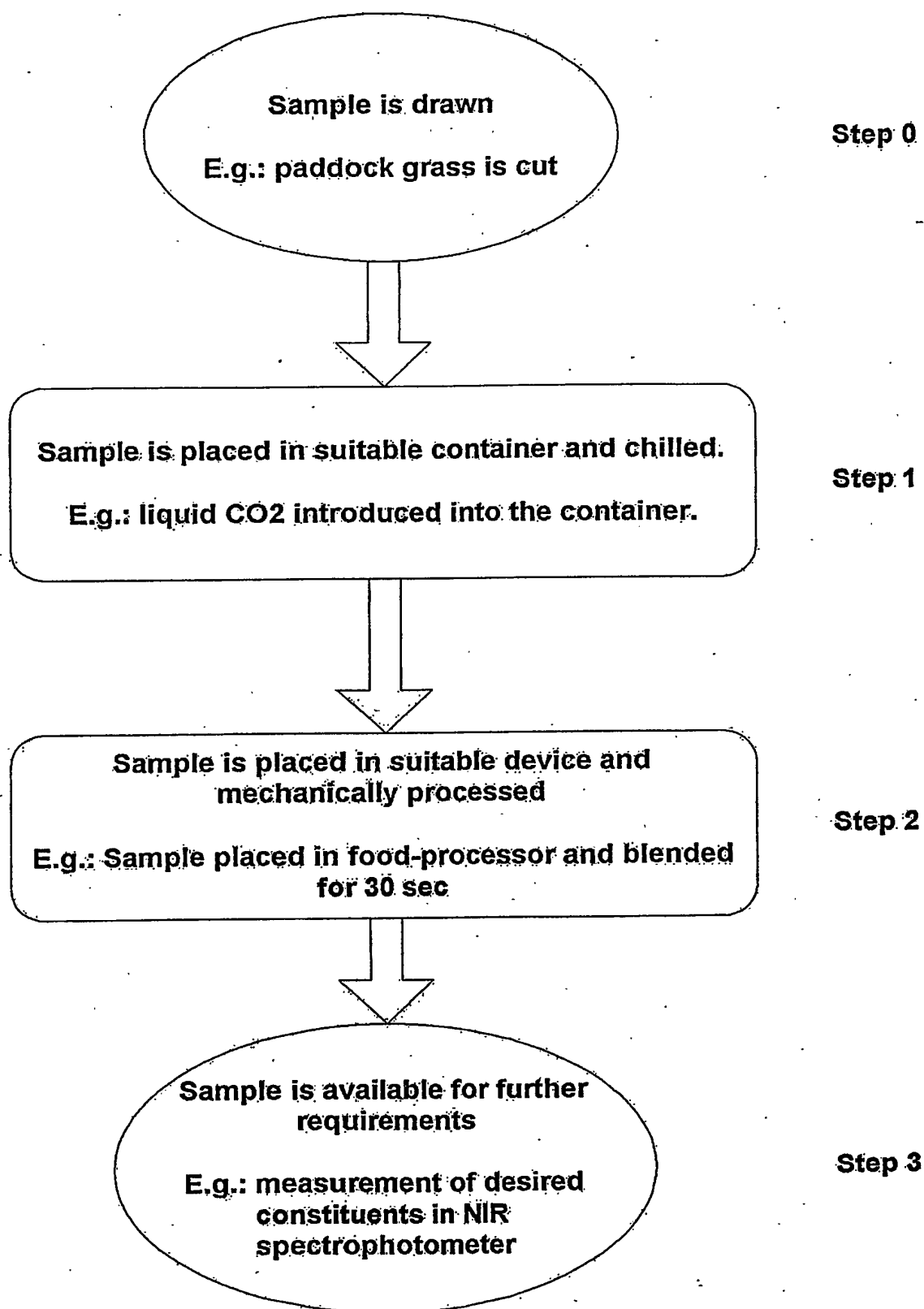


Figure 1



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